

THERMAL DECOMPOSITION OF SALADIPURA PYRITES

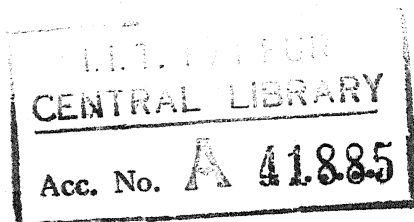
A Thesis Submitted
In Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

By
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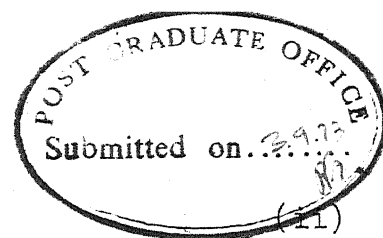


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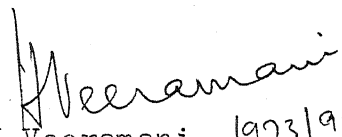
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CERTIFICATE

It is certified that this work 'THERMAL DECOMPOSITION OF SALADIPURA PYRITES' has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

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THERMAL DECOMPOSITION OF SALADIPURA PYRITESABSTRACT

Thermal decomposition experiments using Saladipura pyrites containing 34 percent sulfur were conducted in a fixed bed reactor ^{for} the following range of variables: time 0.5 - 2.0 hr, temperature 450-650°C, particle size 0.69 - 2.0 mm. Complete recovery of labile sulfur was achieved in experiments at 550°C for 0.69 mm particles for reaction periods of 60, 90 and 120 minutes. An optimum temperature level of 550°C was observed and upto 90 percent of labile sulfur was recovered in a reaction period of 1.0 hour. Lower particle sizes would give higher yields of sulfur. Thermal decomposition of pyrites follows a first order rate kinetics with an activation energy of 26.5 kcal/g mole over the temperature range 450-550°C.

CHAPTER I

CHAPTER 1

INTRODUCTION

The annual worldwide consumption of sulfur has been steadily increasing to meet the requirements of present day fertilizer, sulfuric acid and other sulfur based industries. All sulfuric acid plants in India depend on imported sulfur; 85 percent of sulfur used is consumed by the sulfuric acid plants (Table 1.1). Present and anticipated indigenous sources of sulfur are listed in Table 1.2. It is expected further to exploit the use of pyrite deposits by a factor of 4 to 5 by 1975-76. Iron pyrite deposits are found at Amjhore, Bihar and Saladipura, Rajasthan. Potential deposits are estimated at 470 million tons (1); at Amjhore about 300 million tons of deposits have been proved. Pyrite deposits have also been found near Chitaldurg in Mysore (2) and estimated reserves are about 1.5 million tons containing 20-30% sulfur. The Saladipura pyrite deposits are located in Sikar district, Rajasthan. The reserves are estimated to be around 120 million tons of pyrites containing an average of about 25 percent sulfur. The sample used in this investigation contained 34% sulfur. Recovery of elemental sulfur and sulfur dioxide from these pyrite deposits would greatly help in reducing the amount of sulfur imported. This work deals with the recovery of elemental sulfur from Saladipura pyrite ores by thermal

TABLE 1.1SULFUR UTILIZATION IN INDIA (3)

Product	S Requirement (1000 tons)	
	1970-71	1975-76
H ₂ SO ₄	792	1485
Sugar	23	34
CS ₂	26	48
Rubber	5	9
Miscellaneous	96	140
	<u>942</u>	<u>1716</u>

TABLE 1.2SOURCES OF SULFUR (3)

Source	Estimated Supply (1000 tons)	
	1970-71	1975-76
Pyrites	84	350
Zinc and Copper Smelters	30	100
Refinery and Coke Oven Gas	5	15
Imported Sulfur	<u>823</u>	<u>1251</u>
	<u>942</u>	<u>1716</u>

decomposition. The recovery of elemental sulfur by thermal decomposition was studied in a fixed bed reactor and the yield of sulfur obtainable was established for the following operating variables - temperature, time and particle size of the ore.



CHAPTER II

CHAPTER 2

REVIEW OF PREVIOUS WORK

Sulfur can be recovered from pyrites either in the form of elemental sulfur or sulfur dioxide. One half of the sulfur in the pyrite ore is labile and can be recovered by thermal decomposition. The other half of the sulfur in the residual ore can be reclaimed as sulfur dioxide by oxidation.

Sulfur combines directly with almost all elements except gold, platinum and noble gases and exhibits oxidation states from -2 to +6. The principal oxidation states of combined sulfur are -2, 0, +4 and +6 (Table 2.1) (4). The table shows that compounds of sulfur with negative oxidation states can be oxidized to obtain elemental sulfur whereas compounds with a positive oxidation state must be reduced to obtain sulfur in its elemental form. The thermal decomposition of iron pyrite can be considered to result in formation of elemental sulfur and iron sulfide (FeS). The elemental sulfur formed can be recovered easily in an atmosphere devoid of oxygen. However, if S and FeS are left behind to contact with hot air, both the sulfur compounds can be easily oxidized to sulfur dioxide. Thus it would be necessary to maintain an atmosphere relatively free of oxygen in developing a process for recovery of elemental sulfur alone. Air oxidation of Saladipura pyrites in a fixed bed reactor was studied by Ramana Rao (3). Preliminary thermal decomposition studies (3) using Saladipura pyrites showed that an increase

TABLE 2.1PRINCIPAL OXIDATION STATES OF SULFUR (4)

Oxidation State	Group	Example
+6	H_2SO_4	Sulfuric acid
+6	$\text{SO}_4^{=}$	Sulfate ion
+6	SO_3	Sulfur trioxide
+4	H_2SO_3	Sulfurous acid
+4	$\text{SO}_3^{=}$	Sulfite ion
+4	SO_2	Sulfur dioxide
+2	H_2SO_2	Hyposulfurous acid
0	S	Sulfur
-2	H_2S	Hydrogen sulfide
-2	$\text{S}^{=}$	Sulfide ion.

in temperature (500-600°C) and period of decomposition (30-90 min) increases the yield of sulfur obtained in a fixed bed reactor. The reaction of interest can be represented by Equation (1).



In the ~~Orkla~~ process (5) a mixture of copper pyrites and coke is fed to a shaft furnace and heated to 500-760°C and elemental sulfur and gases such as SO₂, H₂S, CS₂, CoS are recovered equivalent to 90% of the sulfur in the original ore. The Noranda process (6) combines the thermal decomposition and air oxidation in a controlled atmosphere so that the exothermic heat released in oxidizing a portion of the pyrite can be used for thermal decomposition; this process would give nearly equal proportions of sulfur and sulfur dioxide (40-50% each).

Outokumpu process (7) utilises a flash smelter at 1250°C and hot combustion gases relatively free of oxygen recovering approximately 47% of the sulfur available as elemental sulfur, the iron pyrrhotite in the residue is oxidised in a fluidized bed roaster recovering an additional 40% of sulfur as sulfur dioxide. Steam roasting of pyrites (8) at 1100-1200°C recovers sulfur in its elemental form and as H₂S and SO₂. The latter two gases can be combined in a Claus reactor to yield a further quantity of elemental sulfur; the sulfur ^{yield} is reported to be about 90%. Sheritt Gordon Mines process (3) decomposes iron

pyrites at 650°C to artificial pyrrhotite and elemental sulfur. The pyrrhotite is further oxidized in an aqueous medium to hydrated iron oxide and elemental sulfur. The total sulfur recovery by this process is estimated at 90%. The salient features of the several processes described above are summarized in Table 2.2.

Literature references give only a brief qualitative description of the many commercial processes listed above. There is very little data available for design purposes. A few references are available which report kinetic rate data for a limited range of conditions. Schwab and Philinis (9) have studied thermal decomposition (600-650°C) of iron pyrites in an inert atmosphere (carbon dioxide) by following the weight loss of a sample during the course of a run. Rajamani and Satyanarayana (10) have investigated the recovery of labile sulfur from Amjhore pyrites in fixed and fluidized beds. The experiments were conducted by these authors in a current of hot air and have reported rate constants assuming the main reaction to be $\text{FeS}_2 = \text{FeS} + \text{S}$ for the temperature range 400-650°C; they used the weight loss method and sulfur content of the ore and residue in getting kinetic data.

It is very unlikely that thermal decomposition occurred in their experiments in a current of air. The extent of oxidation of sulfur and iron sulfide to sulfur dioxide is not included in the interpretation of their data. Ganguly and

TABLE 2.2

SALIENT FEATURES OF ELEMENTAL SULFUR
RECOVERY PROCESSES

Process	Principle	S recovered %
Orkla (5)	Thermal decomposition 500-760°C	90% S, H ₂ S, SO ₂
Noranda (6)	Controlled oxidation + Thermal decomposition 600°C	40% S 50% SO ₂
Ontokumpu (7)	Thermal decomposition with hot gas 1250°C + Fluidized bed roasting	40% S
Steam roasting (8)	1100 - 1200°C Thermal decomposition and Claus reaction	90% S
Sheritt Gordon Mines (3)	Thermal decomposition 650°C + Oxidation in aqueous medium, calcination and reduction	90% S

Banerjee (11) have studied the recovery of sulfur from Amjhore iron pyrites at 900-1000°C by steam, hydrogen and carbon monoxide in a fixed bed reactor at 900-1100°C and recovered sulfur in its elemental form and as H_2S and SO_2 . The contribution of thermal decomposition toward total elimination of sulfur from pyrites was determined to be 50% of total sulfur in pyrite sample in an inert atmosphere (N_2) at 900-1000°C. These studies show that sufficient reliable kinetic data are not available in the published literature for the thermal decomposition reaction.

Several chemical treatment processes have been proposed and tested to recover sulfur from the ore body by solvent extraction. Carbon disulfide being the best solvent for sulfur has often been suggested for extraction of sulfur from ore. Some plants in western countries are based on the use of this solvent but the high cost of solvent, high solvent losses and flammability are its serious disadvantages. Other solvents tried include caustic solution, ammonium sulfide, xylene, kerosene etc. The sulfur can be recovered by either volatilizing the solvent or by crystallization. Chemical treatment processes are not considered in this investigation.

CHAPTER III

CHAPTER 3

EXPERIMENTAL SET-UP

A sketch of the apparatus is shown in Fig.3.1. The major units of set-up are reactor, preheater, temperature controlling devices and condenser.

The reactor is made of 2.5 cm. diameter, 35 cm long S.S. pipe. Nichrome wire (22 gauge, $40\ \Omega$) is wound around the middle of the S.S. tube to heat the ore to desired temperature. Cement and asbestos rope were used to insulate the reactor. The reactor tube was connected to the assembly with a S.S. union joint which permits withdrawal of residue from the reactor after each experiment. A S.S. socket was provided on the side of the reactor for a thermocouple. Chromel Alumel thermocouple wire was used to measure the temperature of the ore body. Lindberg Heavy-Duty furnace was used to preheat the nitrogen gas. Nitrogen gas flow was arranged for down ward flow to carry the sulfur vapors to the condenser below the reactor. Temperature of furnace was controlled by Lindberg Hevi-Duty Pyrometer. Temperature of reactor was controlled by a variac and indicated by Minimate temperature control.

Glass condenser was used to condense the sulfur vapors. The upper end was connected to the reactor bottom with asbestos tape gasket. To the lower end of the condenser a one liter two necked flask, immersed in water bath was attached. N_2 -gas escaped to the atmosphere from the flask.

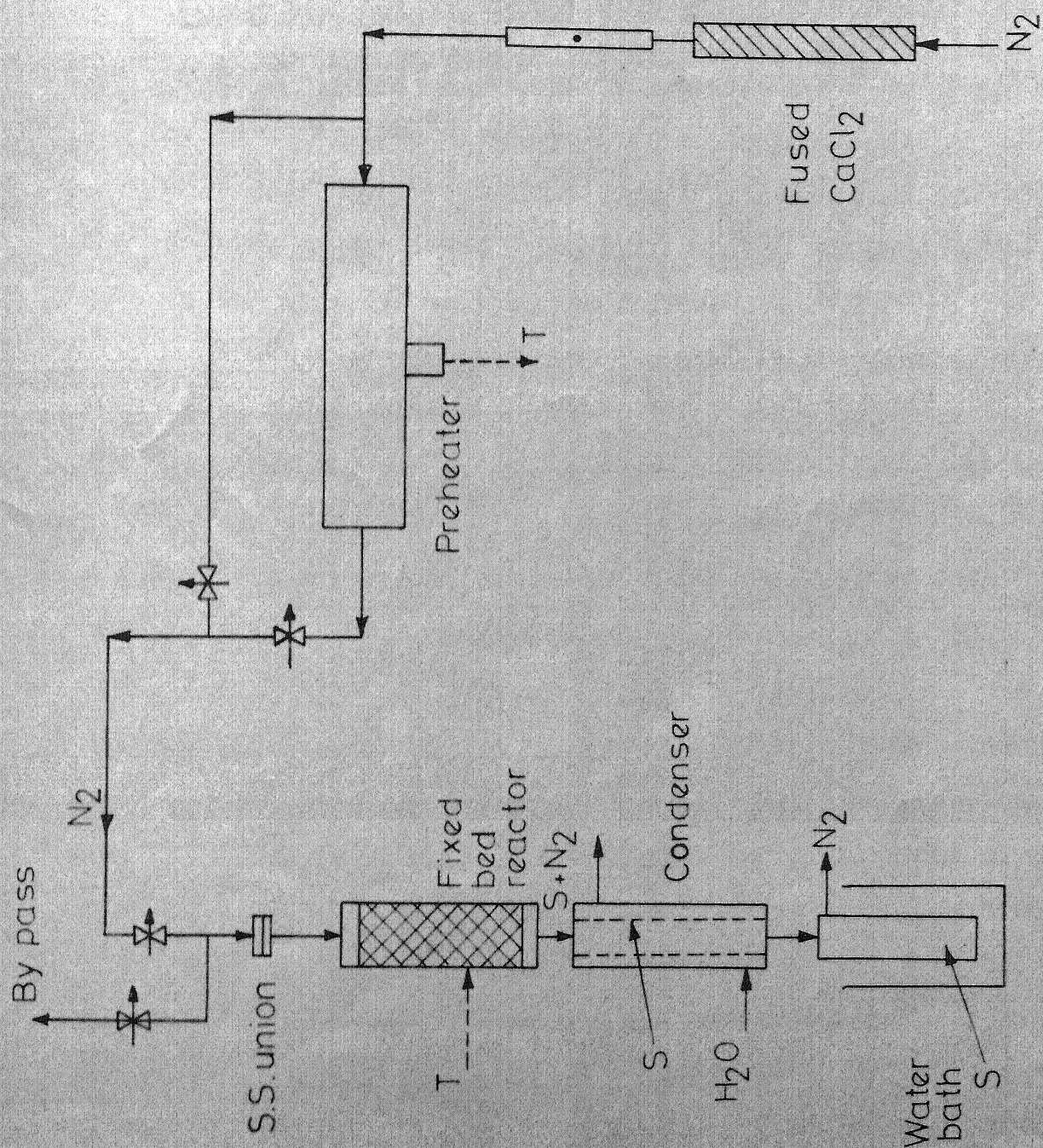


Fig.3.1-Experimental setup for thermal decomposition of pyrites.

"Voltage constants" the voltages at which the inert porcelain beads bed could attain the desired temperature level were determined. The porcelain beads are packed in the reactor upto a height of 16 cm over which was placed 20 grams of the ore sample. Then again beads upto a height of 16 cm. were packed. Wire gauge is put at the bottom to support the bed. The thermocouple, inserted into the middle of the ore body, was connected to the minimate temperature recorder.

The bed was heated to the desired temperature maintaining the rate of heating uniform for all the runs. Once the bed attained the desired temperature, the ore sample was introduced in the reactor and nitrogen flow started.

The time of decomposition for the various runs was counted from the moment the ore sample was introduced to the reactor and brought upto the desired temperature level. After the completion of an experiment, the bed was allowed to cool and the reactor removed from the assembly for withdrawal of the residue. The contents of sulfur in the original ore and residue were determined by procedure described in Appendix A. Sulfur deposited on the walls of the condenser and flask was collected by scrapping and using carbon disulfide. The weight of elemental sulfur collected was determined after evaporation of the solvent.



CHAPTER IV

CHAPTER 4

RESULTS AND DISCUSSION

The thermal decomposition experiments for iron pyrites were conducted for the following range of operating variables.

Temperature of decomposition, T = $450^{\circ} - 650^{\circ}\text{C}$

Reaction Time, θ = 30 to 120 min.

Ore size, D_p = 0.69 - 2.00 mm

The available ore was of -8 mesh and the particles of sizes S_1 (-8 +10 mesh), S_2 (-10 to 20 mesh), S_3 (-20 + 30 mesh) and - 30mesh were separated. The experiments were carried out for the particles of first three sizes because the particles of -30 mesh size could not be supported over the wire mesh in the reactor.

Preliminary runs showed that decomposition of iron pyrite starts around 450°C . It was also found that the amount of sulfur collected per unit weight of ore decomposed was less at about 700°C as compared to that at 550°C . Hence the experiments were planned for the temperature range 450° to 650°C . Reaction period was in the range 30 to 120 minutes based on preliminary exploratory runs.

Sulfur Recovery:

Initially 50 g samples of the ores was used in the experiment. The sulfur collection posed a problem and there

was leakage of sulfur vapors from the system. This problem was overcome by reducing the sample size to 20 g for the latter experiments. The compositions of the ore and residue and the reaction conditions used are summarized in Table 4.1. The Table also reports values for the amount of sulfur collected in each run. The yield of sulfur was determined by a sulfur balance using the initial and final analysis and sample and residue masses. These two values agree very well for most of the runs, the agreement was within 5% and in some upto 15%. For experimental runs 7, 11, 12 and 16 the large deviations are due to accidental breakage of the condenser. The small deviations may be attributed to the small quantities remaining on the walls and inaccessible portions and to some extent losses from the system. The effects of particle size, temperature and time on the recovery of sulfur by thermal decomposition are shown in Figures 4.1, 4.2 and 4.3 respectively. The ordinate represents the yield of sulfur based on sulfur available in the original ore sample.

Particle Size:

Figure 4.1 shows the effect of particle size on the yield of sulfur for experiments conducted for 0.5 to 2.0 hours at 550°C (based on runs 4-6, 10-15 and 20-22). The yeild of sulfur increases from 39% to about 50% for the change in particle size from S_1 to S_3 for 60 minutes of decomposition time.

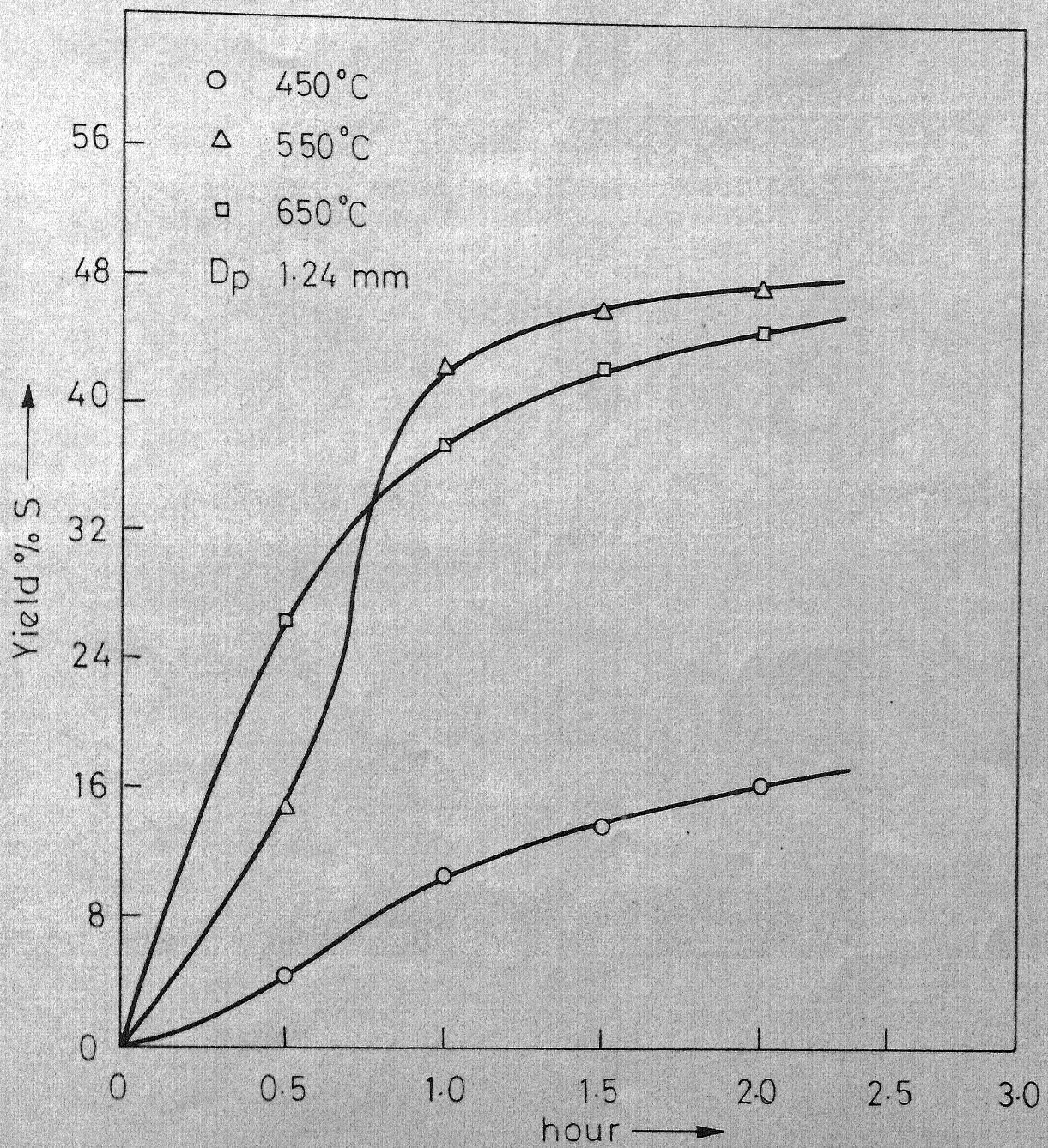


Fig.4.1 - Sulfur yield vs. reaction time (temperature-parameter).

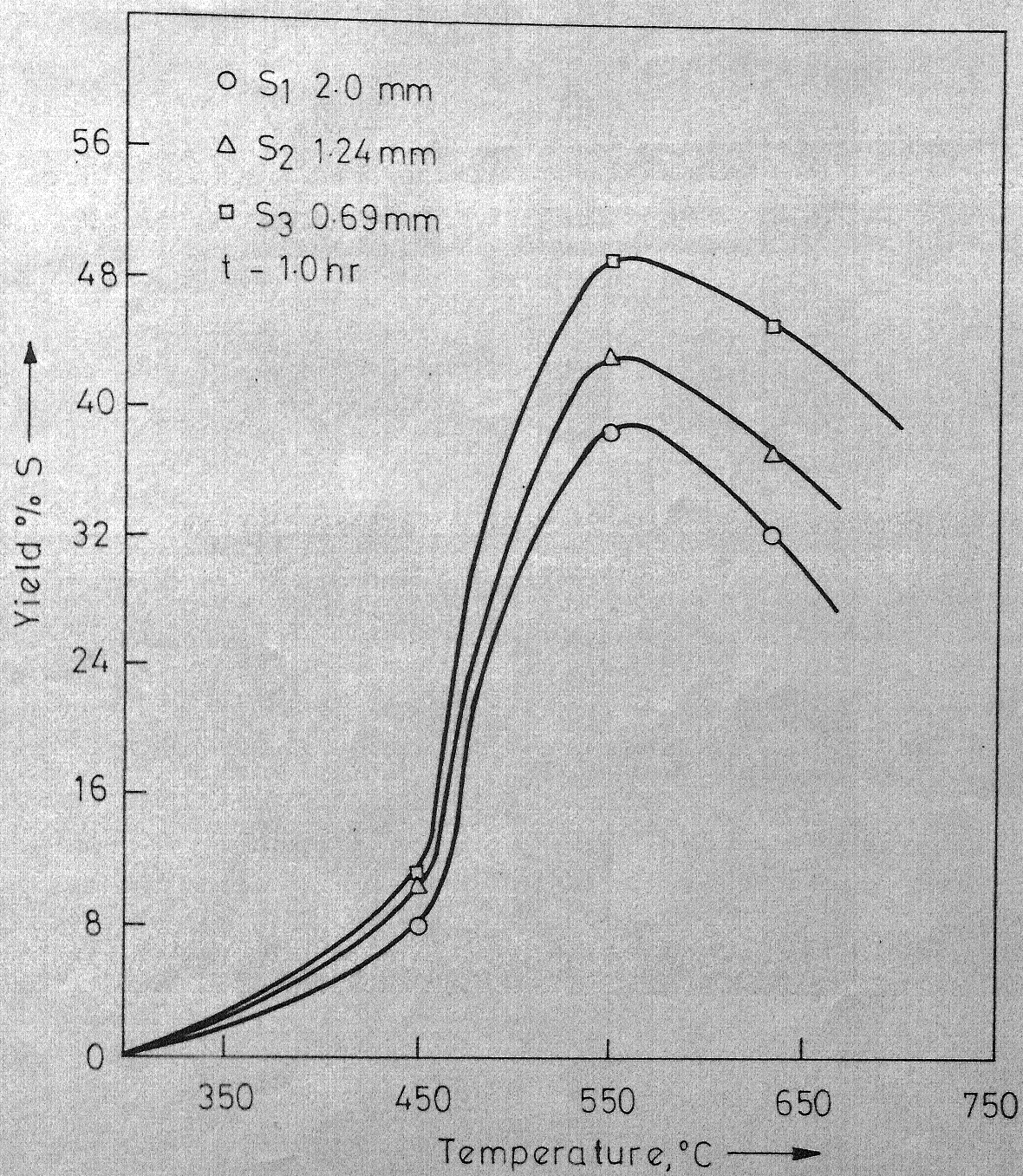


Fig.4.2-Sulfur yield vs. temperature (D_p-parameter).

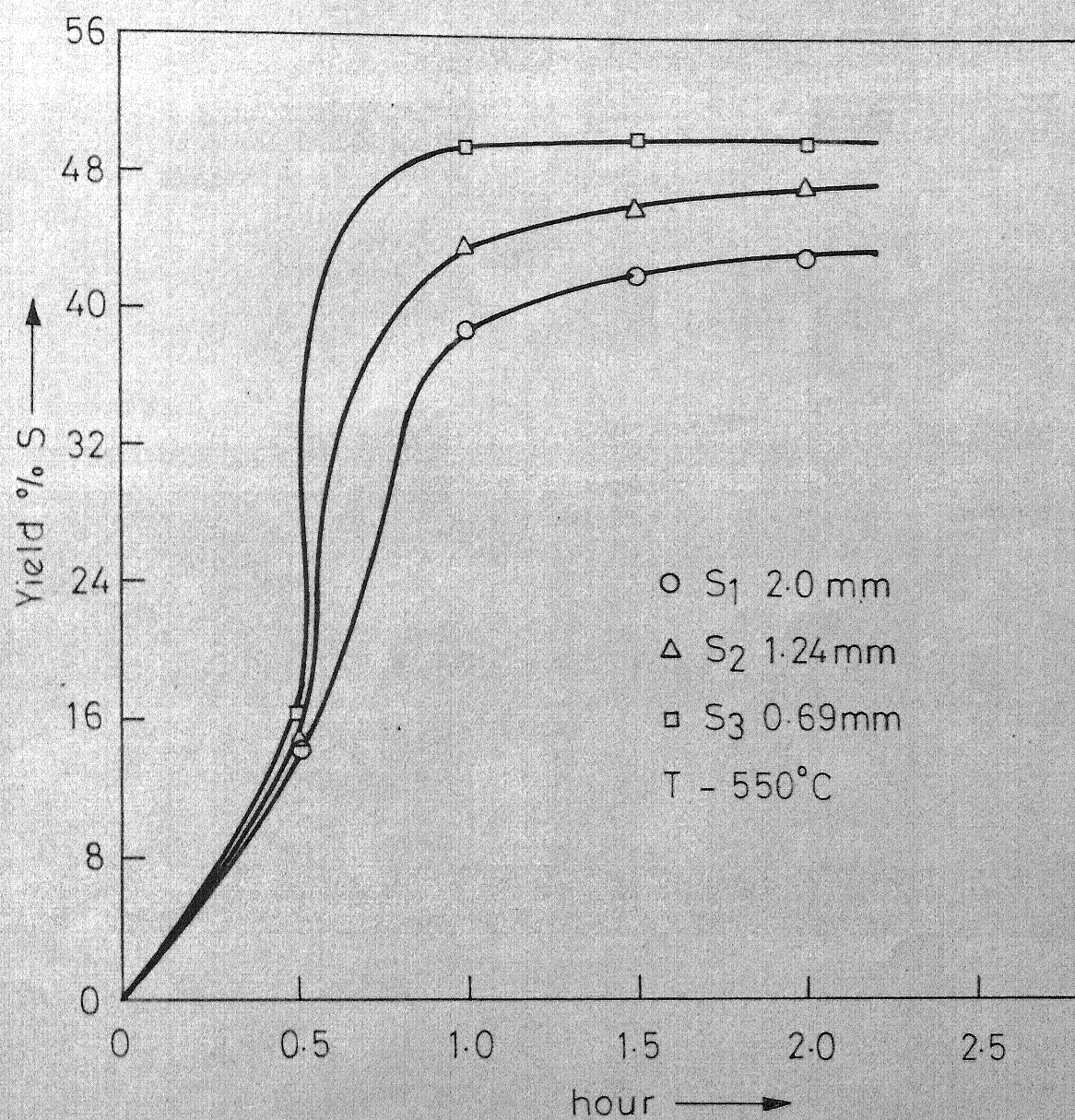


Fig.4.3- Reaction time vs. sulfur yield (D_p-parameter).

TABLE 4.1

EXPERIMENTAL DATA FOR SULFUR YIELD

R.No. (1)	Sample (g) (2)	Pore* size (3)	Temperature °C (4)	Time (min) (5)	Residue (g) (6)	S.Collectd (g) (7)	% S in Residue (8)	Yield of S**	
								Y ₁ (9)	Y ₂ (10)
1	50	S ₁	450	60	48.45	1.05	32.15	6.20	8.20
2	50	S ₂	450	60	48.00	1.60	31.70	9.40	10.60
3	50	S ₃	450	60	47.95	1.70	31.50	10.00	11.20
4	50	S ₁	550	60	43.40	-	24.00	-	38.80
5	20	S ₂	550	60	16.90	2.73	22.80	40.20	43.50
6	20	S ₃	550	60	16.00	3.15	21.40	46.40	49.70
7	50	S ₁	650	60	44.30	-	26.00	-	32.40
8	20	S ₂	650	60	17.40	2.30	24.70	33.80	37.80
9	20	S ₃	650	60	16.50	3.00	22.40	44.20	45.60
10	20	S ₁	550	30	18.90	0.85	30.70	12.50	14.70
11	20	S ₂	550	30	18.80	0.60	30.90	8.85	14.70
12	20	S ₃	550	30	18.60	0.60	30.60	8.85	16.20
13	20	S ₁	550	90	17.00	2.72	23.20	40.00	42.10
14	20	S ₂	550	90	16.72	2.94	21.90	44.00	46.20
15	20	S ₃	550	90	16.44	3.12	20.70	45.80	50.00

Table 4.1 (contd)

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
16	20	S ₂	450	30	19.50	-	33.40	-	4.40
17	20	S ₂	650	30	18.10	1.50	27.60	22.10	26.40
18	20	S ₂	450	90	19.00	0.87	33.00	12.80	13.80
19	20	S ₂	450	90	17.00	2.60	23.00	38.20	42.60
20	20	S ₁	550	120	17.00	2.70	21.70	39.70	43.30
21	20	S ₂	550	120	16.00	2.80	21.60	41.30	47.40
22	20	S ₃	550	120	16.50	3.00	20.60	44.20	50.00
23	20	S ₂	450	120	18.70	1.00	30.40	14.70	16.50
24	20	S ₂	650	120	16.40	2.60	22.90	38.30	44.80

Original ore composition = 34.0% sulfur

*S₁=2.00 mm, S₂=1.24mm S₃=0.69mm

**Y₁=(Sulfur collected/Sulfur in ore)x100 Y₂=Yield based on ore and residue analysis

Nitrogen flow rate was held constant at 5.26 lit/min for all the runs

The large deviations in sulfur yield for Runs 7, 11, 12 and 16 are caused by accidental breakage of glass condenser.

Similar yield increase can be noted for a reaction period of 2 hours also at the same temperature for the size change from S_1 to S_3 . Thus it can be concluded that a decrease in particle size increases the sulfur yield by increasing the surface area available for reaction and diffusion of sulfur vapors to the exterior of the pyrite particles.

Temperature:

The influence of temperature on the recovery of sulfur is shown in Fig. 4.2 with particle size as parameter, (based on runs 1-9). The experiments in this series were conducted for a reaction period of one hour over the temperature range of 450 to 600°C. The curves in Fig. 4.2 show that an increase in temperature over the range 450-550°C increases the sulfur yield to a maximum at about 550 to 570°C and further increase in temperature to 650°C decreases the sulfur yield. This behaviour can be observed for all the three particle sizes over the temperature range 450-650°C. The decrease in sulfur yield above 550°C may be attributed to some phase changes in the residue since it was found from the experiments that the residue bed from runs at 650°C was much harder than the original ore bed and also the residue bed from experiments at lower temperatures 450-550°C. This prevents the escape of labile sulfur from the ore body.

Time:

The influence of time (0.5 to 2.0 hr) on the yield of

sulfur is shown by the curves in Fig. 4.3 with temperature as parameter. The results are based on runs (1,2,5,8,11,14,17,18,19,21,23,24) for a constant particle size S_2 . The results at 550°C show that the recovery of sulfur is 42% for a reaction period of one hour and increased to 48% over a 2.0 hour period. In other words, a reaction period more than one hour results in a small increase (5-6%) only in sulfur yield at the three temperature levels included in this study. These results show that a reaction period of about one hour at 550°C would recover 85-90% of the labile sulfur in the ore as elemental sulfur.

Kinetics and Rate Equation:

The order of the pyrite decomposition reaction was determined by assuming that the decomposition of FeS_2 can be expressed as an irreversible first order or second order reaction with respect to FeS_2 . The reaction rate constants were calculated for a first order and a second order rate mechanism. The values of the rate constants are given in Tables 4.2 and 4.3. It is evident from these tables that the variation in the values of k for the first order reaction were less as compared to those for the second order mechanism. Therefore the final rate equation was assumed to be of first order with respect to FeS_2 . Schwab and Philinis (9), Rajamani and Satyanarayana (10), and Ganguly and Banerjee (11) have also reported the decomposition reaction to be of the first order.

TABLE 4.2
FIRST ORDER RATE CONSTANTS FOR THERMAL DECOMPO-
SITION OF PYRITES

R.No.	Temp. °C	Time (min)	Ore * Size	Moles FeS ₂ (n _{A0})	Moles FeS ₂ (n _A)	k(hr ⁻¹)
16	450	30	S ₂	0.106	0.0966	0.183
2	450	60	S ₂	0.265	0.2088	0.236
18	450	90	S ₂	0.106	0.0766	0.214
23	450	120	S ₂	0.106	0.0710	0.202
11	550	30	S ₂	0.106	0.0748	0.702
5	550	60	S ₂	0.106	0.0135	2.076
14	550	90	S ₂	0.106	0.0080	1.728
21	550	120	S ₂	0.106	0.0052	1.548
17	650	30	S ₂	0.106	0.0498	1.512
8	650	60	S ₂	0.106	0.0280	1.338
19	650	90	S ₂	0.106	0.0154	1.290
24	650	120	S ₂	0.106	0.0106	1.170
10	550	30	S ₁	0.106	0.0748	0.696
4	550	60	S ₁	0.265	0.0620	1.464
13	550	90	S ₁	0.106	0.0166	1.248
20	550	120	S ₁	0.106	0.0140	1.014
12	550	30	S ₃	0.106	0.0716	0.7878
6	550	60	S ₃	0.106	0.0005	5.3820
15	550	90	S ₃	0.106	0.0000	-
22	550	120	S ₃	0.106	0.0000	-

Table 4.2 (contd.)

1	450	60	S_1	0.265	0.2212	0.1836
3	450	60	S_3	0.265	0.2056	0.2338
7	650	60	S_1	0.265	0.0930	1.0440
9	650	60	S_3	0.106	0.0090	2.4840

* $S_1 = 2.00$ mm $S_2 = 1.24$ mm $S_3 = 0.69$ mm

TABLE 4.3

SECOND ORDER RATE CONSTANTS FOR THERMAL DECOMPO-
SION (TRIAL CALCULATIONS)

R.No.	Temperature (°C)	Time (hr)	$k \text{ hr}^{-1} \text{ mole}^{-1}$
16	450	0.5	1.90
2	450	1.0	1.02
18	450	1.5	2.40
23	450	2.0	2.33
11	550	0.5	7.80
5	550	1.0	64.55
14	550	1.5	77.03
21	550	2.0	91.27
17	650	0.5	21.30
8	650	1.0	26.35
19	650	1.5	37.03
24	650	2.0	42.52

The values of k at various temperatures were obtained by plotting $\ln (n_{Ao}/n_A)$ against time and fitting a least squares straight line to the experimental data (Figures 4.4 to 4.7). The average rate constants obtained from these figures are given in Table 4.4. The variance and standard deviations for these are shown in Table 4.5. The other values of k included in Table 4.4 are taken from Table 4.2. These values are compared with the results of earlier investigators (10,11,12) in Table 4.4. Direct comparison with the results of Rajamani and Satyanarayana(10) is not possible since their results do not isolate the thermal decomposition and air oxidation effects. The results of Ganguly and Banerjee (11) fall outside the range of this investigation. The value of k reported by Schwab and Philinis (9) was for a particle size of 0.1 mm whereas this work utilized particle size range 0.69 to 2.0 mm. It can be concluded from the values in Table 4.4 that the k values of this investigation are of the same order of magnitude as those reported by the earlier workers.

Effect of Temperature:

Dependence of the rate constant ' k ' against the temperature has been shown in the Fig. 4.8, (three points corresponding to particle size S_2 at 450, 550 and 650°C and the fourth one corresponds to particle size S_1 at 550°C) A straight line was fitted by the method of least squares. The slope of the line gives the activation energy of the order of $18.2 \frac{\text{Kcal}}{\text{gmole}}$. The

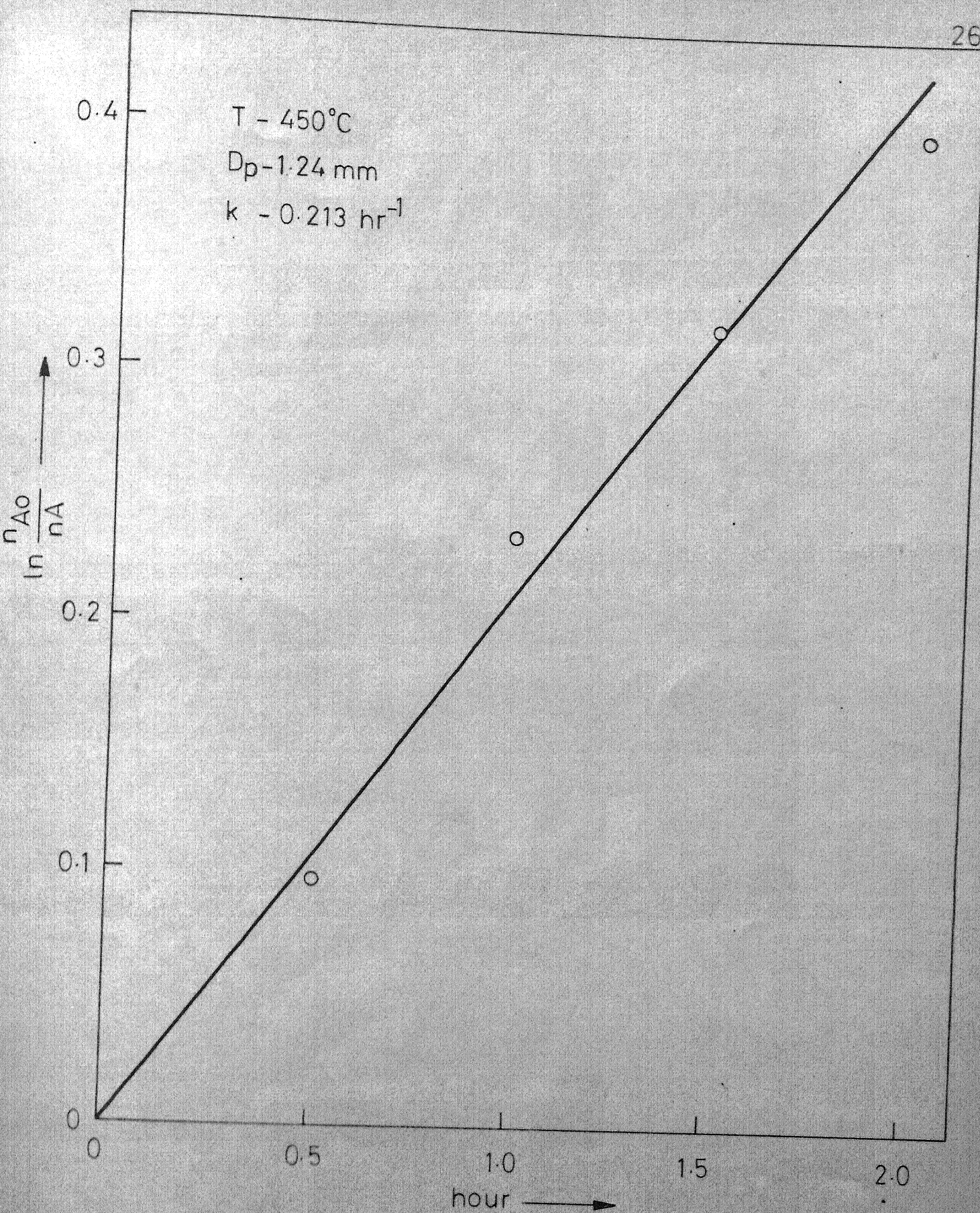


Fig 4.4 - Rate data at 450°C .

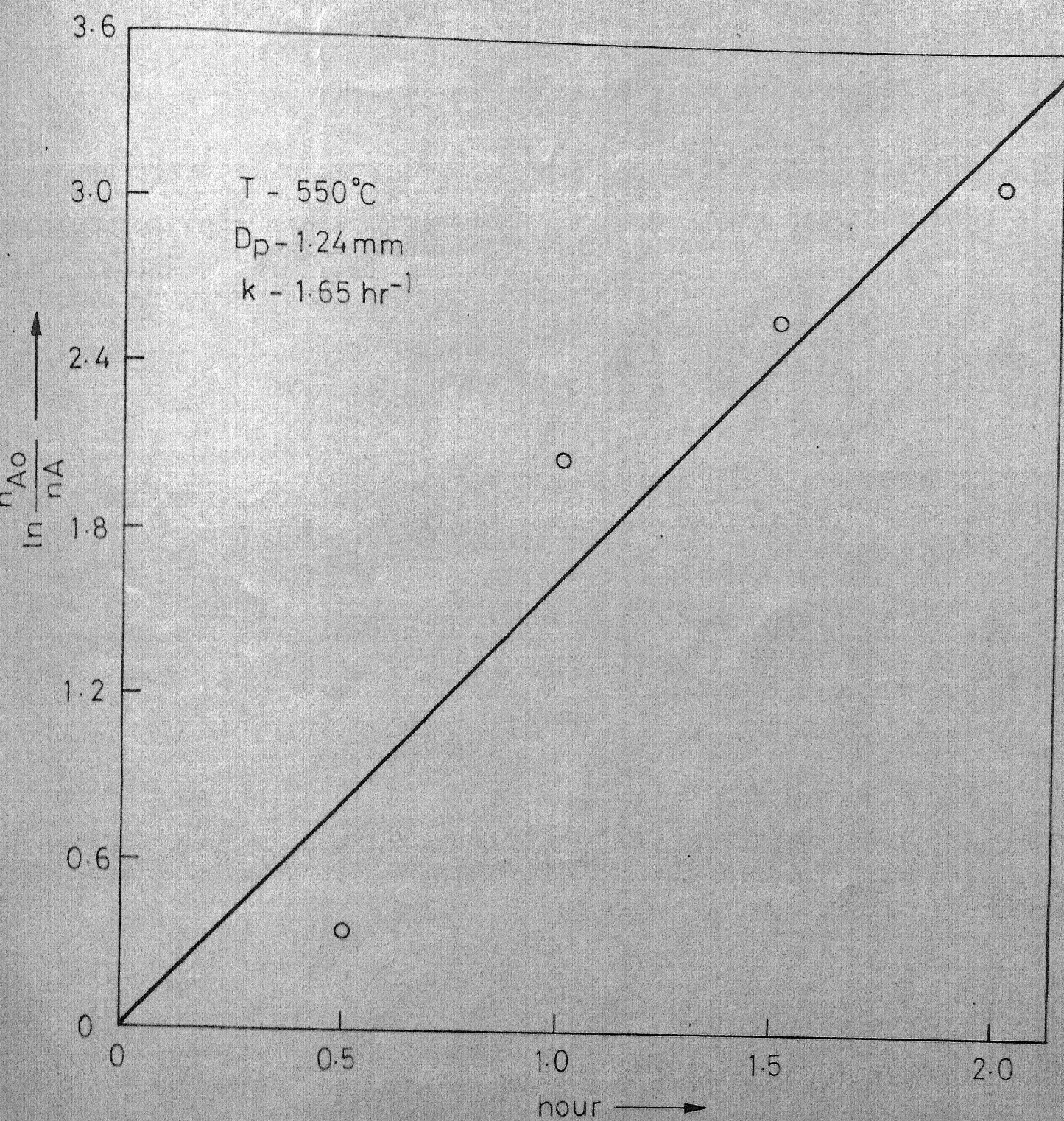
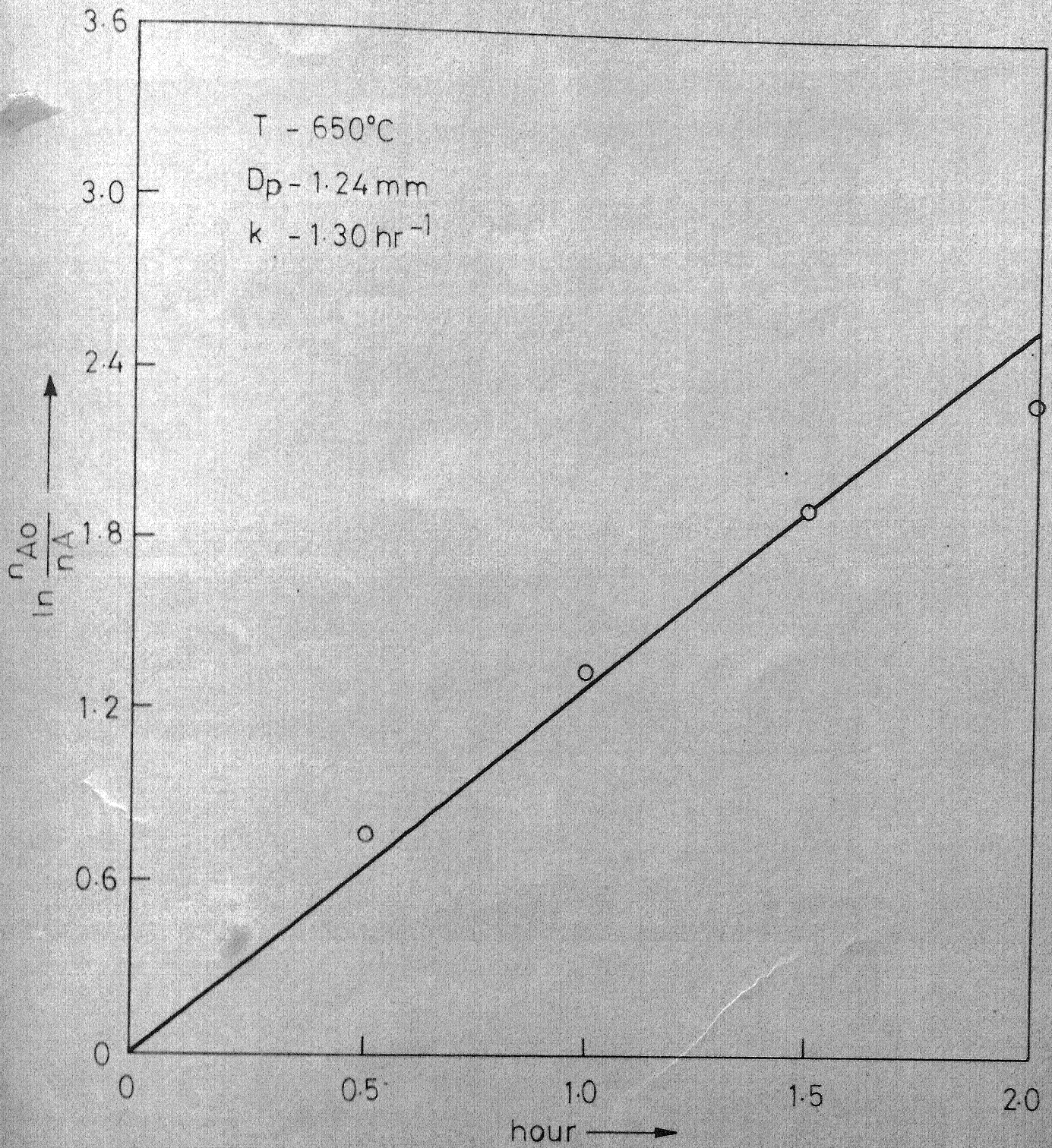


Fig.4-5 - Rate data at 550°C.

Fig.4.6 - Rate data at 650°C .

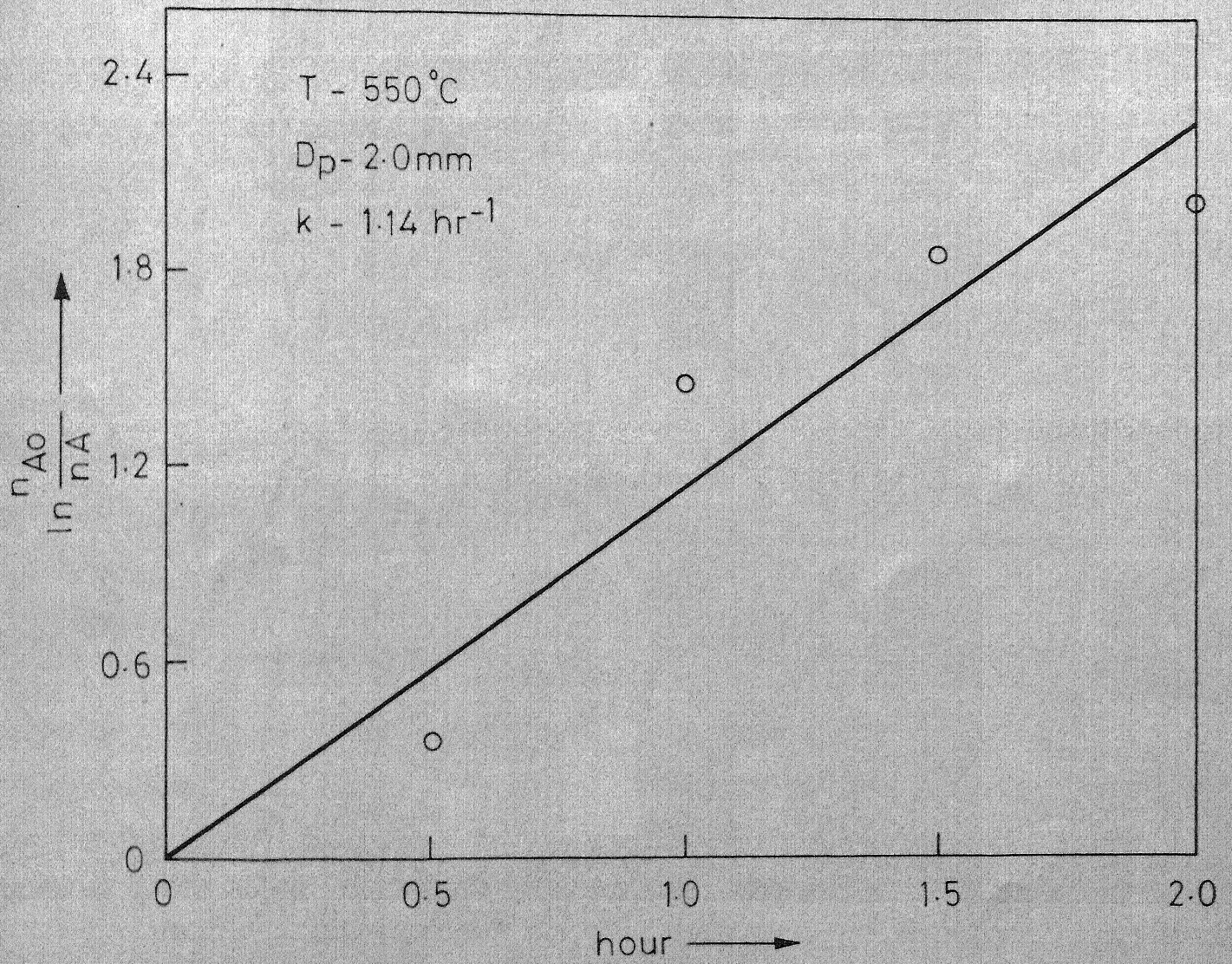


Fig.4.7-Rate data at 550°C .

TABLE 4.4

COMPARISON OF FIRST ORDER RATE CONSTANTS* FOR THERMAL
DECOMPOSITION

Temp., °C	Present Study		Rajamani (11)			Schwab(10)	Ganguly (12)	
	0.69mm	1.24mm	2.00 mm	0.50mm	1.50mm	2.00mm	0.10mm	0.13mm
400	-	-	-	0.233	6.140	0.242	-	-
450	0.234 ^a	0.213 ¹	0.184 ^d	0.954	0.630	0.369	-	-
550	5.382 ^b	1.650 ²	1.140 ⁴	8.292	2.410	1.900	-	-
650	2.484 ^c	1.300 ³	0.044 ^e	20.640	8.580	8.340	2.54	-
900	-	-	-	-	-	-	-	1.76
1000	-	-	-	-	-	-	-	2.56

*_{hr}⁻¹ ^aRun 3 ^bRun 6 ^cRun 9 ^dRun 1 ^eRun 7¹Fig.4.4 ²Fig. 4.5 ³Fig.4.6 ⁴Fig. 4.7

TABLE 4.5

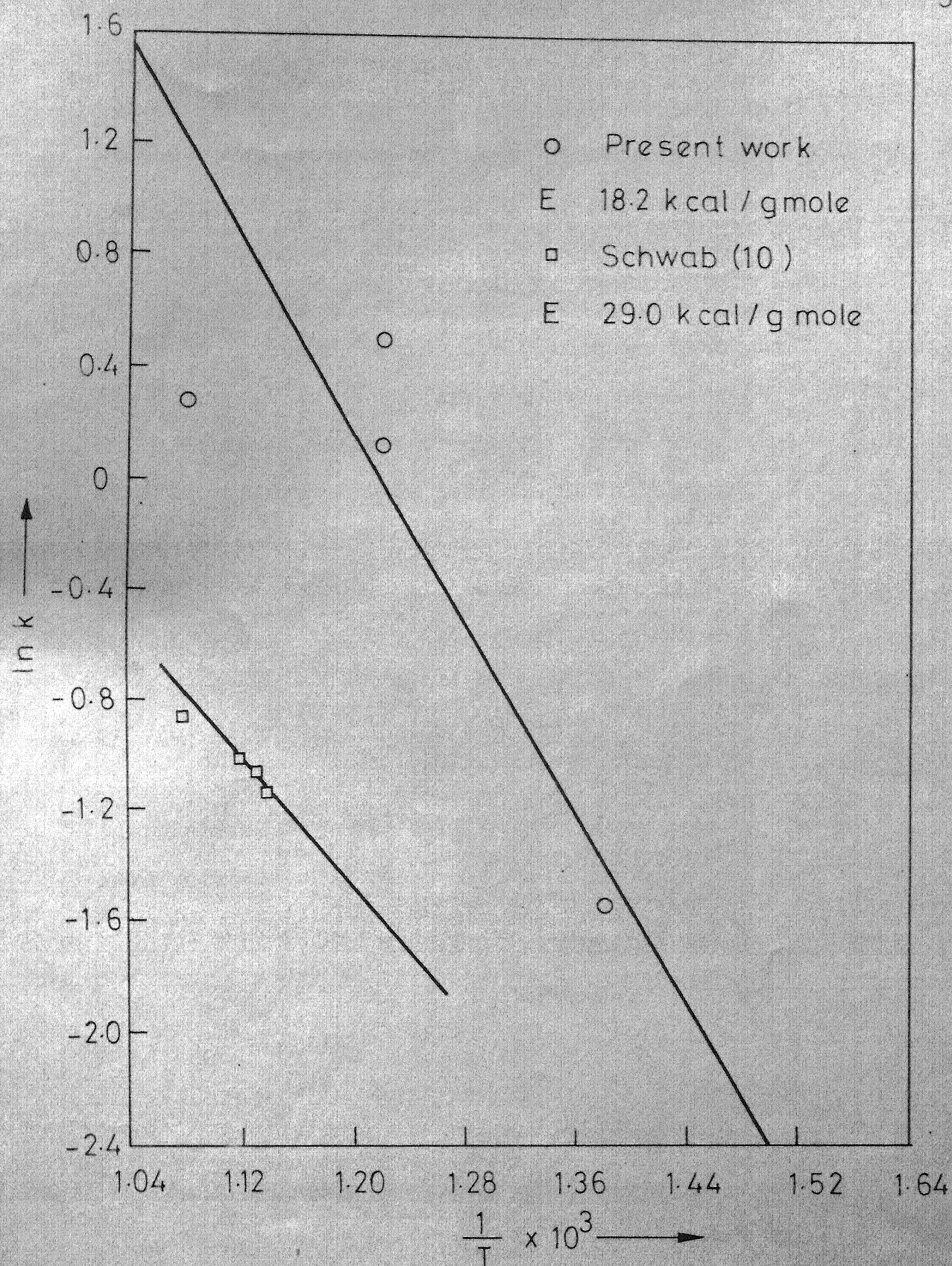
LEAST SQUARE FIT OF EXPERIMENTAL DATA

Run No.	$\ln \frac{n_{Ao}}{n_A}$	$\ln \frac{n_{Ao}}{n_A}$ (graphical)	Deviation	Standard deviation	Variance
16	0.097	0.106	-0.009		
2	0.236	0.213	0.023		
18	0.321	0.320	0.001	0.017	0.02×10^{-2}
23	0.402	0.426	-0.024		

11	0.350	0.820	-0.470		
5	2.075	1.650	0.425		
14	2.590	2.480	0.110	0.337	11.3×10^{-2}
21	3.100	3.300	-0.200		

17	0.755	0.640	0.115		
8	1.337	1.280	0.057		
19	1.935	1.920	0.015	0.150	2.24×10^{-2}
24	2.303	2.580	-0.277		

10	0.348	0.56	-0.212		
4	1.460	1.140	0.320		
13	1.865	1.700	0.165	0.244	5.94×10^{-2}
20	2.030	2.280	-0.250		

Fig.4-8 - Plot of $\ln k$ vs. $1/T$.

activation energy was also calculated neglecting the point at 650°C and found to be the order of 26.5 Kcal/g.mole. This value is nearer to the value of activation energy 29 Kcal/g.mole reported by Schwab (9). Schwab's (9) results are also plotted in Fig. 4.9 in the range of 600-650°C.

It is clear from Fig. 4.8 that the data obtained at 650°C are not in conformity with the data obtained at 450 and 550°C. This is because of the formation of harder residue bed at 650°C as compared to that at 450° and 550°C. It may be concluded that the decomposition of FeS_2 is a first order reaction with respect to FeS_2 with activation energy of the order of 26.5 Kcal/g.mole in the temperature range of 450-550°C. Therefore this rate expression would be adequate for design calculations of pilot plant studies (Appendix B).

The combined effect of all the variables studied viz temperature, particle size and reaction time on the sulfur yield were correlated by the following equation:

$$Y = -510.56 + 1.70 X_1 + 0.92X_2 + 13.30 X_3 + 0.26 X_3^2 - 0.04X_1X_3$$

Where X_1 = Temperature of ore body, °C

X_2 = Period of decomposition, min.

X_3 = Particle size, mm

Y = Sulfur expelled/Sulfur originally present

The above equation was regressed by means of least square technique. The calculated and observed values were compared in Table 4.6.

TABLE 4.6

CORRELATION OF EFFECTS OF TIME, TEMPERATURE AND
PARTICLE SIZE ON YIELD OF SULFUR

Run. No.	Y*	Y ₂	%Deviation
1	10.24	8.20	24.90
2	11.62	10.60	9.47
3	12.80	11.20	10.35
4	35.12	38.80	-9.54
5	39.18	43.50	-9.95
6	42.32	49.70	-14.85
7	32.09	32.40	- 0.96
8	38.85	37.80	-2.85
9	43.94	45.60	-3.68
10	17.76	14.70	20.40
11	21.82	14.70	48.50
12	24.96	16.20	54.20
13	42.97	42.10	20.70
14	47.04	46.20	18.15
15	50.17	50.00	0.34
16	-4.99	4.40	-
17	20.75	26.40	-21.50
18	18.73	13.80	35.70
19	47.45	42.60	11.35
20	41.32	43.30	-4.48
21	45.39	47.40	-4.25
22	48.52	50.00	-2.96
23	16.34	16.50	-1.00
24	46.55	44.80	3.28

$$*Y = -510.56 + 1.70X_1 + 0.92 X_2 + 13.30 X_3 + 0.26X_3^2 - 0.04X_1X_3$$

X₁ = Temperature, °C

X₂ = Time, Min.

X₃ = Particle Size, mm

Y₂ = Yield based on ore and residue sulfur analysis



CHAPTER V

CHAPTER 5

CONCLUSIONS

The following conclusions can be made from this study:

1. Complete recovery of labile sulfur has been achieved in experiments at 550°C for 0.69 mm particles for reaction periods of 60, 90 and 120 minutes.
2. An optimum temperature level of 550°C has been established.
3. Upto 90 percent of the labile sulfur can be recovered in a reaction period of 1.0 hour.
4. Lower particle sizes give higher yields of sulfur.
5. The thermal decomposition of iron pyrites follows first order rate kinetics with an activation energy of 26.5 kcal/g mole in the temperature range of 450-550°C.
6. The results of this investigation can be fruitfully utilized for the design of pilot plant reactor for the thermal decomposition of pyrites.

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APPENDICES

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APPENDIX ADETERMINATION OF TOTAL SULFUR IN PYRITE OREPrinciple:

A known quantity of finely powdered dried ore is oxidized to sulfate on treatment with sodiumperoxide followed by leaching with conc. HCl and sulfuric acid produced is precipitated as barium sulfate.

Procedure:

Two grams of the finely powdered ore sample in a weighing tube is dried at 50°C for one hour to remove the moisture. About 0.5 gm of the sample from the weighing tube is weighed and put in nickel crucible (50 ml capacity) containing a layer of one gram of fusion mixture (equal amounts of Na_2CO_3 and K_2CO_3). The sample in the crucible is mixed with fusion mixture and 2-3 gms. of sodium peroxide added and the contents mixed. The mass in the crucible is covered with an even layer of sodium peroxide to absorb SO_2 evolved. The crucible is put in a muffle furnace at 500°C for half an hour. The crucible with the fused mass is put into hot water in a 500 ml. beaker and leached with concentrated HCl and sulfuric acid is produced. The solution in the beaker is boiled for about five minutes, cooled and then filtered into 500 ml volumetric flask using Whatman filter paper no.40. Pipette 50 ml of the solution in the volumetric flask and add

200 ml distilled water and 15 ml of liquid ammonia to preipitate the hydroxides of the elements of group IIIB (Fe, Al, Cr) present. Beaker contents are boiled to remove the excess ammonia. Addition of concentrated HCl dissolves the hydroxides of Fe, Al, Cr. About one gram of hydroxyl amine hydrochloride is added to convert the -ous-ions into -ic ions, to prevent co-precipitation of iron sulfate (Here iron is much which can affect the sulfur determination) on treatment with Barium chloride. The contents of the beaker are then boiled and 15 ml of 10% (W/V) BaCl_2 solution is added and BaSO_4 is precipitated. The precipitate is allowed to settle for about 4 hours and filtered through Whatman filter paper no.42. The precipitate is washed with hot distilled water till the chloride is removed (chloride test is carried out with silver nitrate solution).

The filter paper with precipitate is put into a silica crucible and charred for 1-2 hours in a muffle furnace (at about 1000°C). The temperature of the furnace is increased gradually to prevent spurting of the precipitate. The crucible is taken out of the furnace and cooled in a desicator. The residue after cooling is weighed as Barium sulfate.

$$\text{Percent sulfur in sample} = \frac{B \times f}{A}$$

where, B = weight of sample taken

A = weight of the residue

f = factor to calculate the quantity of sulfur in BaSO_4
(13.74)

APPENDIX B

PILOT PLANT DESIGN FOR THERMAL DECOMPOSITION OF PYRITES

Laboratory scale thermal decomposition studies of Saladipura pyrites in a fixed bed reactor have shown that about 90 percent of the labile sulfur in pyrites can be recovered as elemental sulfur in an inert atmosphere at an optimum temperature level of 550-570°C for a reaction period of one hour. Thermal decomposition reaction follows a first order rate expression with an activation energy of 26.5 Kcal/g mole. It would be desirable to avoid exposure of the Saladipura pyrites to a temperature in excess of 600°C due to the formation of a hard residue bed leading to lower sulfur recovery. A fluidized bed reactor would be better suited to maintain a uniform bed temperature.

The following units would comprise the pilot plant set-up for thermal decomposition of Saladipura pyrites.

- ore preheater (fluidized bed)
- fluidized bed reactor
- sulfur condenser
- Indane combustion

A sketch of the proposed apparatus is shown in Fig-B-1(a). The ore feed arrangement is shown in Fig. B-1(b). Heat requirements of the process are obtained by the combustion of Indane using minimum excess air. Supplemental electrical heating of the preheater and reactor walls can also be provided. It would be necessary to use minimum excess air so that the combustion gases

used in the reactor would have less oxygen which would reduce the yield of elemental sulfur due to oxidation to sulfur dioxide.

The reactor is fed with dry fine preheated pyrite ore particles. The ore bed is fluidized with the help of hot flue gases coming after burning Indane gas. The sulfur vapors generated by thermal degradation of iron pyrites are condensed in water cooled condenser to give liquid sulfur product. The gases from the condenser may be combined with a portion of the combustion gases and used to preheat the ore in a fluidized bed unit.

Design of Pilot Plant Apparatus:

Preliminary design of the major units in the pilot plant apparatus is given below:

Basis: sulfur production 5kg/hr.

Raw material: Ironpyrite (34%S)

Particle size: 0.69 mm (assumed uniform)

Fluidized Bed Reactor

A four fold decrease in reaction period for the roasting of pyrites in a fluidized bed reactor in comparison to fixed bed unit was observed by Rajamani (10). This would suggest a reaction period of 15 minutes for this study in a fluidized bed reactor. However a conservative estimate of 25 minutes is used for the preliminary design to allow for the differences in thermal decomposition and roasting.

Rate constant of the fluidized bed reactor can be obtained from:

$$n_{A_0}/n_A = e^{-k_1 \tau_1} = e^{-k_2 \tau_2} \quad (B-1)$$

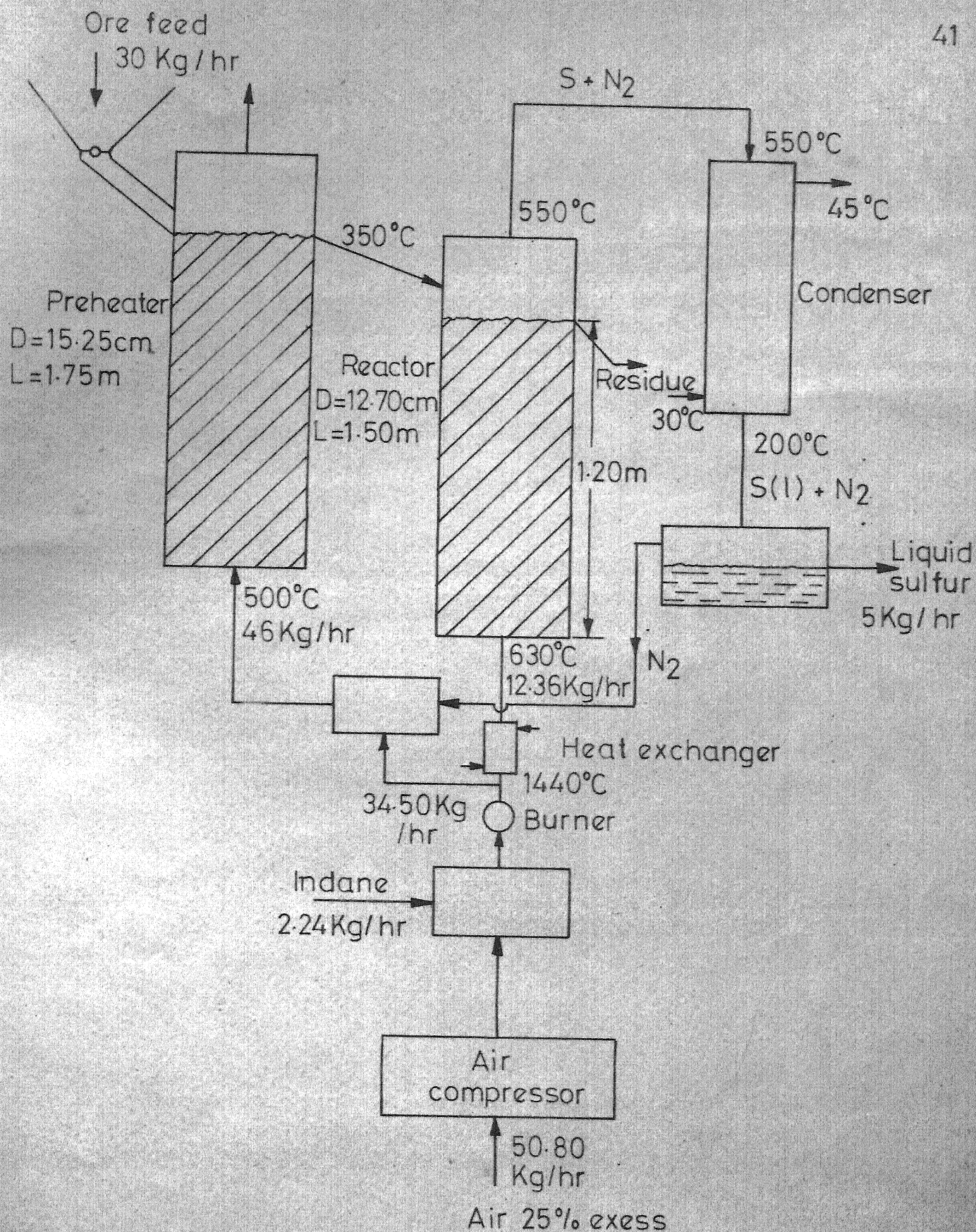
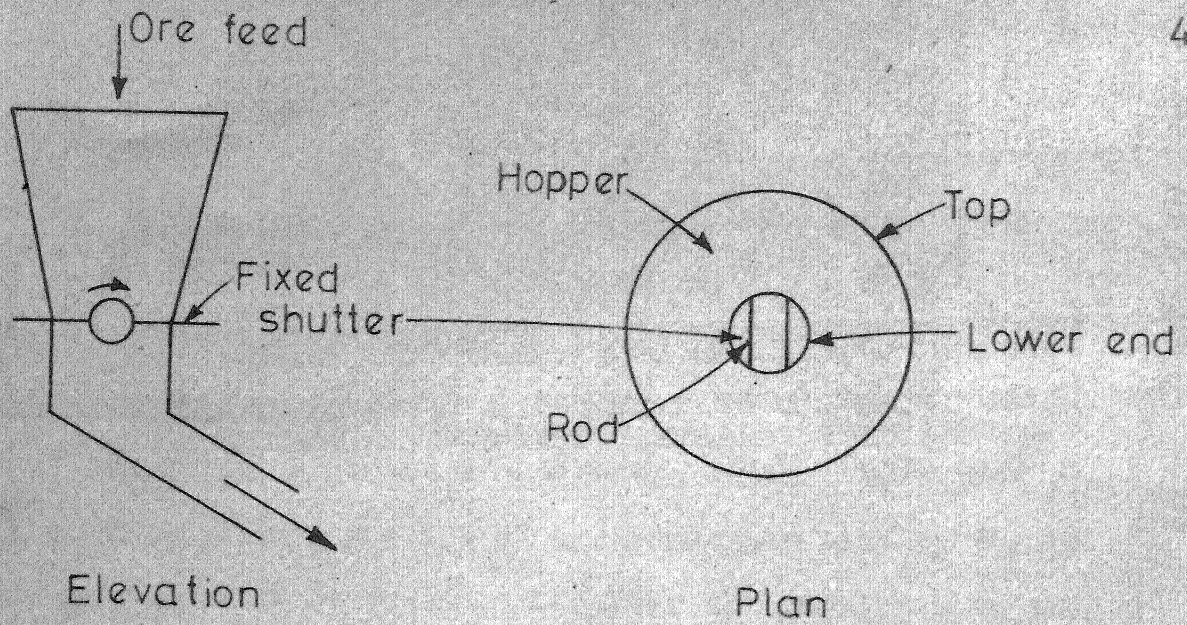
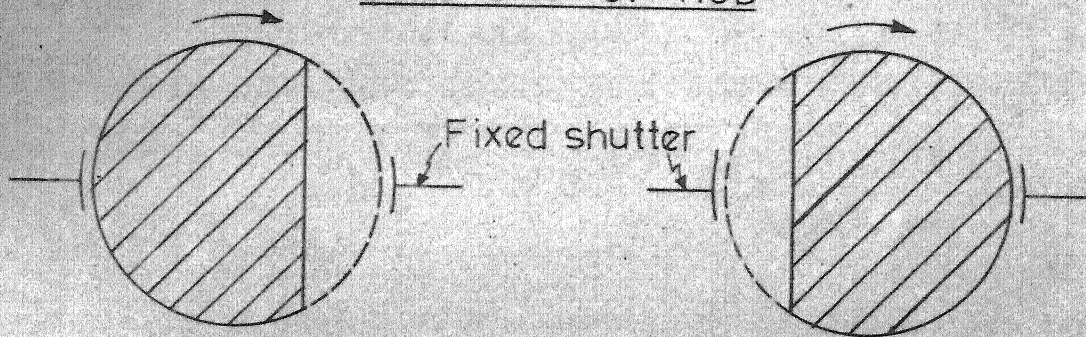


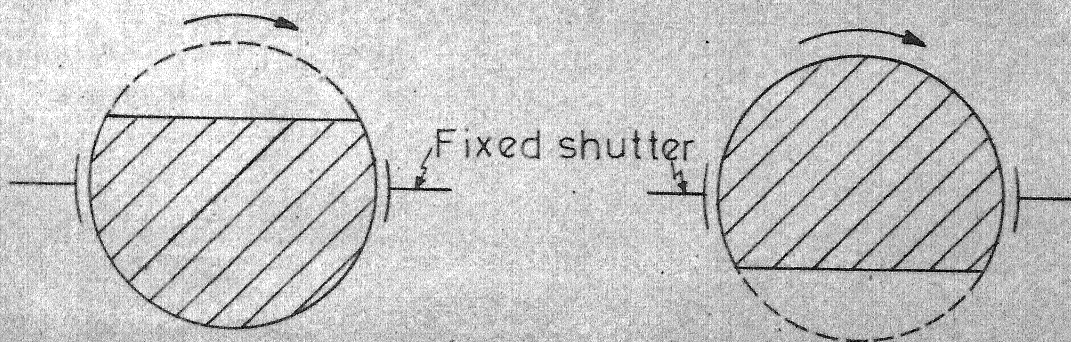
Fig. B1(a) - Pilot plant design.



SECTIONS OF ROD



a. Material being fed.



b. Material not being fed.

Fig. B 1 (b) - Feed arrangement for the ore.

where k_1, k_2 and τ_1, τ_2 indicate first order rate constants and reaction periods for fixed and fluidized beds respectively.

For $k_1 = 5.30 \text{ hr}^{-1}$; $\tau_1 = 60 \text{ minutes}$, $\tau_2 = 25 \text{ minutes}$
Eq.(B-1) gives $k_2 = 12.9 \text{ hr}^{-1}$

Average Conversion (\bar{x}_B):

In a fluidized bed reactor the conversion, x_B of reactant in a single particle depends on its length of stay in the bed. However, length of stay is not same for all particles in the reactor hence we must calculate average conversion of the material.

Thus for back mix flow of the single size of solid particles which is completely converted in time τ we obtain

$$1 - \bar{x}_B = \int_0^{\tau} (1 - x_B) e^{-t/\bar{\tau}} dt \quad (\text{B-2})$$

where $\bar{\tau}$ = average residence time.

The result of the present study showed that decomposition of pyrite was reaction rate controlled and that rate could be expressed by a first order reaction with respect to moles of FeS_2 given by $n_{A_0}/n_A = e^{-kt} = 1 - x_B$ (B-3)

$$\begin{aligned} (1 - \bar{x}_B) &= \int_0^{\tau} e^{-kt} e^{-t/\bar{\tau}} dt \\ \text{or } (1 - \bar{x}_B) &= \frac{1 - e^{-(k + \frac{1}{\bar{\tau}})\tau}}{\bar{\tau}(k + \frac{1}{\bar{\tau}})} \quad (\text{B-4}) \end{aligned}$$

Average Residence Time:

Values of \bar{x}_B are calculated from eqn.(B-4) for different assumed values of $\bar{\tau}$ as in the following table. An average residence time of $\bar{\tau} = 35 \text{ minutes}$ is chosen for an average conversion of 88.1%.

\bar{t} min.	\bar{x}_B (%)
10	68.2
20	81.1
30	86.6
35	88.1
50	90.85
80	94.5

Minimum Fluidization Velocity* - Gmf

Viscosity of hot gases mainly N_2 (600°C)	$\mu = 0.0087$ cp
Density of solid particles, ρ_s	$= 5.0$ g/cc
Density of N_2 at 600°C, ρ_F	$= 0.391 \times 10^{-3}$ g/cc
Particle size, $D_p = .69$ mm	$= 0.0272$ inches
$(\rho_s - \rho_F) \rho_F$	$= 0.757 \left(\frac{lb}{cuft} \right)^2$
Minimum fluidization velocity using monograph*, G_{mf}	$= 50$ lb/hr sq.ft.
	$= 0.0244$ Kg/hr cm^2

Reactor Size:

FeS_2 Feed (n_{A_0})	$= \frac{5}{32 \times 0.881}$	$= 0.177 \frac{kg \text{ mole}}{hr}$
		$= 19.24$ kg/hr
Wt. of ore feed (63.7% FeS_2)		$\frac{19.24}{0.673} = 30.2$ Kg/hr
Volume of ore ($\rho_{bulk} = 1.63$ g/cc) bed		$= 18.5 \times 10^3$ cc
Volume of average residence of 35 minutes		$= 10.8 \times 10^3$ cc

The recommended L/D ratio for a fixed bed is 8-10. This would give a fixed bed height of 85.5 cm for a diameter of 12.7 cm (5 in.)

Hence height of fixed ore bed $= 85.5$ cm.

Diameter of reactor $= 12.7$ cm.

*Max Leva 'Chem.Engg. Series Fluidization' page 67, 1959.

Expanded bed height chosen, L = 1.4 meters

Total height = 1.5 meter

R = L/85.5 = 1.64

For R = 1.64 and Dp = 0.027 inch $\frac{G_f}{G_{mf}} = 4^*$

$G_f = 0.0244 \times 4 \text{ Kg/hr sq.cm.}$

$= 0.0244 \times 4 (12.7)^2 \pi / 4 = 12.36 \text{ Kg/hr.}$

Assuming G_f as 5.0%O₂ 11.0%CO₂ 84% N₂

Sp. heat at 600°C (g.cal/g.mole °C) 7.62 11.05 7.23

Heat given by hot gases

$$= 12.36 \left(\frac{0.84}{28} \times 7.23 + \frac{0.11}{44} \times 11.05 + \frac{0.05 \times 7.62}{32} \right) \times (630-550)$$

$$12.36 \times 0.2554 \times 80 = 253.0 \text{ Kcal}$$

$$\text{Heat taken by pyrite ore} = \frac{30.2 \times 10^3 \times 21.7}{120} (550-350)$$

$$= 1084 \text{ Kcal}$$

$$\text{Heat of reaction} = 18.6 \times 177.0 = 3290 \text{ Kcal.}$$

$$\text{Electrical heat to be supplied (75\% eff)} = \frac{3290 + 1084 - 253}{0.75}$$

$$= 5500 \text{ Kcal} = 6.4 \text{ Kw hr.}$$

Preheater

G_f (84%N₂, 11% CO₂, 5%O₂)

$$\text{Heat taken by ore} = \frac{30.2 \times 10^3 \times 21.7}{120} (350 - 25)$$

$$= 1760 \text{ Kcal.}$$

Heat given by hot gases

$$= G_f \left(\frac{0.84 \times 7.23}{28} + \frac{0.11 \times 11.05}{44} + \frac{0.05 \times 7.62}{32} \right) (500-350)$$

*Max. Leva 'Chem. Engg. Ser., Fluidization' p.102

$$\text{or } G_f (0.2554) 150 = 1760$$

$$G_f = 46 \text{ Kg/hr}$$

Let diameter of preheater be 6 inches = 15.25 cm.

$$G_{m_f} = 0.0244 \times \frac{(15.25)^2}{4} \pi = 4.48 \text{ Kg/hr.}$$

$$\therefore \frac{G_f}{G_{m_f}} = \frac{46}{4.48} = 10.3$$

$$\text{From Max Leva } \frac{\text{Fixed bed height}}{\text{expanded bed}} = 2.78$$

$$\text{Fixed bed height} = \frac{10.8 \times 10^3}{\pi \times \frac{(15.25)^2}{4}} = 59 \text{ cm.}$$

$$\therefore \text{Expanded bed height} = 59 \times 2.78 = 164 \text{ cm} \\ = 1.64 \text{ meter}$$

$$\text{Total preheater height} = 1.75 \text{ meter}$$

Indane-Air Requirement Excess air = 25%

$$\text{Reactor: } G_f = 12.36 \text{ Kg/hr.}$$

$$N_2 = 12.36 \times 0.84 \text{ Kg/hr}$$

$$\text{Air} = 12.36 \times \frac{0.84}{0.79} = 14.2 \text{ Kg/hr.}$$

$$O_2 = 14.2 \times \frac{0.21}{32} = 0.0932 \text{ Kg mole/hr}$$

Indane is assumed to be pure butane (C_4H_{10})



$$\text{Indane} = \frac{0.0392}{6.5} \times 75 = 0.01075 \text{ Kg mole} = 0.625 \text{ Kg/hr}$$

Prehater

$$m_1 = 12.36 \times 0.95 = 11.5 \text{ Kg/hr}$$

$$m_2 = 46 - 11.5 = 34.5 \text{ Kg/hr}$$

$$\text{Air} = 34.5 \times \frac{0.84}{0.79} = 36.6 \text{ Kg/hr.}$$

$$\text{Indane} = \frac{36.6 \times 0.21 \times 0.75}{32 \times 6.5} = 0.0278 \text{ Kg mole} = 1.61 \text{ Kg/hr.}$$

$$\text{Total air} = 14.2 + 36.6 = 50.8 \text{ Kg/hr.}$$

$$\text{Total Indane} = 0.625 + 1.61 = 2.35 \text{ Kg/hr.}$$

Temperature of Combustion Gases: Heat transfer eff. = 70%

$$\text{Heat of combustion of Indane} = 687.64 \frac{\text{Kcal}}{\text{gmole}}$$

$$(T-25) \left\{ (7.88 + 12.36 \times \frac{0.84}{28} + 34.5 \times \frac{0.84}{28}) + 12.7 \left(\frac{12.36 \times 0.11}{44} + \frac{34.5 \times 0.11}{44} \right) + \left(\frac{34.5 \times 0.05 \times 8.3}{32} \right) \right\} = \frac{2.235}{58} \times 10^3 \times 687.64 \times 0.7$$

$$\text{or } T = 1440^\circ\text{C}$$

Condenser Design

Assume all excess O_2 reacts with S to form SO_2 and calculations done on dry basis

Heat given by hot gases, ΔH_1

	<u>Cp (400°C)</u> <u>g cal/gmole °C</u>	<u>g moles</u>
N_2	7.09	371
CO_2	10.46	35
SO_2	10.94	9.65

$$\Delta H_1 = (371 \times 7.09 + 35 \times 10.47 + 9.65 \times 10.94) 10^{-3} (550-200)$$

$$= 1080 \text{ Kcal}$$

$$\text{Heat of condensation of sulfur vapors} = 68.4 \times \frac{5}{32} \times 10^3 = 10700 \text{ Kcal}$$

$$\begin{aligned} \text{Total heat load} &= 10700 + 1080 \\ &= 11780 \text{ Kcal} \\ &= 13.6 \text{ Kw hr.} \end{aligned}$$

Heat Taken by Cooling Water:

Heat transfer coefficient during condensation (Vertical Tube)

$$h_m = 1.47 \left(\frac{4w}{\pi D \mu_f} \right)^{-1/3} \left(\frac{\mu_f^2}{k_f^3 \rho_f^2 g} \right)^{-1/3}$$

Liquid properties

k_f = Thermal conductivity Btu/hr sq.ft.(°F/ft)

ρ_f = Density of sulfur liquid, $\frac{\text{lb}}{\text{ft}^3}$

w = Mass flow rate of sulfur lb/hr.

μ_f = Viscosity lb/hr.ft.

$\rho_f = 1.808 \text{ g/cc} = 1.808 \times 62.43 \text{ lb/ft}^3$

$k = 330 \text{ Btu/hr sq.ft.}(^\circ\text{F/ft})$

$\mu_f = 2.42 \times 215 \text{ lb/hr.sq.ft.}$

$w = .0027 \times 10^3 \text{ lb/hr.}$

$D = 1 \text{ inch}$

$g = 32.2$

hence we calculate $h_m = 1065 \text{ Btu/hr sq ft}^\circ\text{F}$

Effect of Non-Condensibles

Non condensibles = 72.6%

Assume that sulfur behaves as oil during condensation than Fig.13.17 Kern shown a reduction in h_m to one sixth. Hence affective h_m is given as

$$(h_m)_{\text{eff}} = \frac{1065}{6} = 177.5 \text{ Btu/hr sq ft}^\circ\text{F}$$

Overall heat transfer coefficient

Neglecting the resistance of $h_i(400)$ on U we calculate

$$\frac{1}{U} = \frac{1}{177.5} + \frac{R_o}{R_i} \frac{1}{200} + \frac{1}{400} = \frac{1}{177.5} + \frac{11}{10 \times 200}$$

$$U = 111.0 \text{ Btu/hr sq.ft.}^\circ\text{F}$$

$$= 0.0545 \text{ Kcal/hr.sq.cm}^\circ\text{C}$$

$$(\Delta T_m) = 275$$

$$\text{Hence heat taken by cooling water} = 0.0545 \times 275 \times A = 11780$$

$$A = \frac{11780}{0.0545 \times 275} = 790 \text{ cm}^2$$

$$\text{Diameter of condensing tube} = 2.54 \text{ cm}$$

$$\text{Height of tube} = \frac{790}{2.54} = 105 \text{ cm.}$$

$$= \frac{105}{30.5} = 3.47 \text{ ft.}$$

$$\text{No. of tubes} = 4 \text{ (each tube of 1 ft)}$$

Summary

Reactor:	Height = 1.5 meters Diameter = 12.7 cm (5 inches) Ore feed = 30.2 Kg/hr (350°C) G_f = 12.36 Kg/hr
Preheater:	Height = 1.75 meters Diameter = 15.25 cm (6 inches) Ore feed = 30.2 Kg/hr (25°C)
Condenser:	No. of tubes (1 ft) = 4 Liquid sulfur (200°C) = 5 Kg/hr
Indane and Air required:	Indane = 2.24 Kg/hr Air = 50.8 Kg/hr

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